

# PATENT SPECIFICATION

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## COMPLETE SPECIFICATION.

### Improvements in or relating to The Manufacture of Starch Decomposition Products.

5 We, ESCHER WYSS G.m.b.H., a German Body Corporate, of Ravensburg, Württemberg, Germany, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:

10 This invention relates to a process and installation for the continuous manufacture of starch decomposition products, in which process and installation a chemically and/or biologically active substance is added to a starch suspension, the mixture being passed continuously through a reaction vessel with  
15 temporary acceleration of the flow followed by direct mixing with steam, the added substance becoming active during and directly after the steam passage in a field of action of high imposed stress by impacting and/or shearing forces. We have claimed this process as claim 1 of United Kingdom Patent Application No. 49829/65 (Serial 1,028,212).

20 In United Kingdom Patent Specification No. 995,660 we have disclosed and claimed apparatus which forms a suitable reaction vessel for use in effecting the step of temporarily accelerating the flow followed by direct mixing with steam.

30 Our objects in the present invention are to improve and develop the above process, particularly for producing *d*-glucose or dextrin, by means of additional steps which adjust the required chemical or physical properties of the product, and to provide installations suitable for use in performing the improved process.

35 The present invention consists in a continuous process for the manufacture of starch decomposition products, particularly  
40 *d*-glucose or dextrin, which process comprises the steps of adding a chemically and/or biologically active substance to a starch suspension, passing the mixture continuously through a reaction vessel with temporary acceleration of the flow followed by direct mixing with steam, the added substance becoming active during and directly after the steam passage in a field of action of high imposed stress by impacting and/or shearing forces, and conducting the primarily liquified starch paste thus obtained through at least one additional reaction vessel for the purpose of continuing the degradation reaction and adjusting required chemical or physical properties, the free cross-sectional area of the additional reaction vessel being larger than that of the pipe conduits before and after the first-mentioned reaction vessel.

45 The invention also consists in an installation for effecting the process according to the preceding paragraph, comprising a first reaction vessel for the continuous primary liquefaction of starch as a result of the action of a chemically and/or biologically active substance, means for inactivating or destroying this active substance, and a second reaction vessel for continuation of the degradation reaction, said second reaction vessel being connected between said first reaction vessel and said means and having a free cross-sectional area which is larger than the pipe conduits before and after the said first reaction vessel.

60 The performance of the invention will now be described, by way of example only, with reference to the accompanying drawings in which:—  
65 Figure 1 shows an installation in accordance with the invention for the production of *d*-glucose;

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Figure 2 shows an installation in accordance with the invention for the production of dextrin; and

5 Figures 3 and 4 show the additional reaction vessels for continuing the degradation reaction, on a larger scale.

10 According to Figure 1, the starch suspension is prepared in a tank 1 with a stirrer mechanism 2, mixed with enzyme, and then conveyed by means of a proportioning pump 3 through the installation. Gelatinisation and primary liquefaction of the starch takes place in a first reaction vessel 4. The mixture is conducted through the reaction vessel 4 with a temporary acceleration of flow and subsequent mixing with steam flowing through a pipe 5, the previously added active substances (enzymes) becoming operative during the steam passage and immediately after the continuous starch gelatinisation in an effective field of high imposed stress due to impacting and/or shearing forces. The reaction vessel 4 can for example be the arrangement described in United Kingdom Patent Specification 995,660 and which comprises a tube having one or more constrictions in cross-section through which the starch suspension is conducted and in the wall of which, after the cross-sectional constriction and in the transition range to the full tube cross-section, one or more inlet orifices are provided for the steam.

30 From the first reaction vessel 4, the primarily liquefied starch paste contained therein passes selectively into one of two additional and mainly cylindrical reaction vessels 6<sup>1</sup>, 6<sup>2</sup> for the purpose of continuing the degradation reaction and adjusting required properties, the said paste flowing longitudinally through said vessels, the free-cross-sectional area of which is larger than that of the pipe conduits before and after the first reaction vessel 4.

45 The vessel 6<sup>1</sup> contains screens or sieves 7, 7<sup>1</sup>, 7<sup>2</sup>, 7<sup>3</sup>, 7<sup>4</sup>, which extend at a certain distance from one another over the entire cross-section of the vessel and serve for producing a uniform flow and also for producing a mixing effect. The free-cross-sectional area of the individual apertures in the screens preferably decreases from the inlet end towards the outlet end of the vessel, so that consequently the screen 7<sup>4</sup> of the vessel 6<sup>1</sup> has smaller apertures than the screen 7.

55 The vessel 6<sup>2</sup> contains a displacement member 8 of cylindrical form with semi-spherical ends, the said member being arranged co-axially of the vessel 6<sup>2</sup> and being held by webs 9. There is thus produced an annular cross-section for the free passage of the starch paste. The displacement member 8 also serves to steady the flow. By suitable choice of the diameter and length of the displacement member 8, it is however

also possible for the effective volume of the vessel 6<sup>2</sup> and thus the residence time of the starch paste in the vessel to be modified.

70 In the event that a subsequent saccharification with a temperature-sensitive enzyme is to follow a primary liquefaction, for example by a thermophilic carbohydrase, a reaction vessel 10 of substantially the same type as the reaction vessel 4 is connected on the output side, into which the intermediate product obtained at the outlet of the vessel 6<sup>1</sup> or the vessel 6<sup>2</sup> is introduced. Instead of steam, this secondary active substance is fed as a solution or suspension in water through a pipe 11 into the reaction vessel 10. The reference 12 designates a tank for the preparation of this solution or suspension, while 13 is a stirrer mechanism and 14 is a pump for conveying the solution or suspension into the pipe 11. One or more additional active substances can also be introduced in the same way as the secondary active substance is supplied.

90 In order that the temperature permissible with this secondary active substance may not be exceeded in the starch product, a cooler 15 is also provided in the installation, through which cooler the starch product can if necessary be conducted before it is introduced into the reaction vessel 10. In certain circumstances, however, it is sufficient for the additional active substance to be fed in as a solution or suspension in an appropriate quantity of water for the purpose of cooling the flowing starch product.

100 Connected after the reaction vessel 10 is an additional vessel 16 of the same type as the reaction vessel 6<sup>1</sup> or 6<sup>2</sup>, and serves for the continuation of the degradation reaction. It is however possible for the flowing product, after feeding in the additional active substance, to be also subjected in apparatus 17 to impacting and shearing effects of high frequency for the purpose of thoroughly mixing the components in the form of a film with single or multiple deflection and acceleration. The device involved here can with advantage be an arrangement comprising a high speed rotor formed with toothed rims and a corresponding stator.

115 Finally, for completing the enzyme action, the product obtained is conducted through a device 18 which is similar in construction to the reaction vessel 4 serving for the gelatinisation. This device 18 serves for the thermal inactivation of the active substances. For the purpose of raising the product to a temperature which is advantageously higher than 110°C., the product is mixed in this device with steam under superatmospheric pressure, the said steam being supplied to the device 18 through a pipe 19. Following the device 18 is a pipe conduit 20, in which the heated product is further exposed for some time to the high temperature for the

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purpose of complete inactivation of the active substances. Thereafter, the product is discharged through a throttle member 21 and an expansion chamber 22. In the same way as the thermal enzyme inactivation, it is also possible by means of the device 18 to carry out a chemical enzyme inactivation by feeding in a substance which acts as a poison to the enzyme.

The following two examples are intended to provide further details of the process of this invention as performed using the installation of Figure 1. All percentages are percentages by weight unless otherwise stated:

#### EXAMPLE 1

By mixing 100 kg. of maize starch of normal commercial moisture content with 120 litres of water, a 40% starch suspension is prepared in the tank 1 and 0.3 to 0.5% of  $\alpha$ -amylase of industrial purity, based on the air-dry starch batch is added thereto and the suspension is adjusted to the optimum reaction conditions by adding chemicals. The product is continuously conveyed through the reaction vessel 4 and, by feeding in steam at a pressure of 5 to 10 atm. gauge, and by imposing stress by impacting and/or shearing forces is heated to 95°C., gelatinised and primarily liquefied. For continuing the degradation reaction, the starch solution is pumped through one of the reaction vessels 6<sup>1</sup>, 6<sup>2</sup>, and after reaching a controlled degree of degradation, passes through the reaction vessel 10, which is of similar construction to the reaction vessel 4. The intermediate product is diluted in vessel 10 to 30 to 35% solid content by feeding in a solution or dispersion of  $\beta$ -amylase or  $\alpha$ -glucodiase in cold water from the tank 12, and is thus cooled to 60 to 70°C. and mixed with this second specifically active enzyme. The product is thereafter advantageously conveyed into the device 17 for intensively mixing the enzyme with the primarily produced dextrin solution and subsequently conducted into the vessel 16 for continuing the degradation reaction. Finally, it passes through the device 18, the inactivation zone 20 and the expansion chamber 22. The sugar solution thus obtained is purified, concentrated and either worked up to a syrup with a high maltose or glucose content or transferred into an arrangement (not shown) for the crystallisation of the dissolved sugar.

#### EXAMPLE 2

The production of d-glucose is effected substantially by the same process as that indicated in Example 1. Before admixing a temperature-sensitive enzyme, however, the product initially liquefied by thermophilic  $\alpha$ -amylase is cooled in the cooler 15 to such a degree that the addition of the carbohydrase intended for the subsequent

saccharification can be effected with small quantity of water sufficient for the complete mixing and the final saccharification can be carried out in a temperature range from 40 to 60°C.

The installation according to Figure 2 is intended for the production of dextrin. It comprises in part the same devices and apparatus as the arrangement shown in Figure 1, and consequently these are provided with the same reference numerals.

The starch suspension prepared in the tank 1 and mixed with amylase is again conveyed by means of the proportioning pump 3 into the reaction vessel 4, in which the gelatinisation and primary liquefaction of the starch is effected by mixing with steam supplied from the pipe 5. In this case, instead of the vessels 6<sup>1</sup>, 6<sup>2</sup> for continuing the degradation reaction, mainly cylindrical vessels 6<sup>3</sup>, 6<sup>4</sup> constructed in a particular manner are provided, these vessels being traversed longitudinally by the primarily liquefied starch paste originating from the reaction vessel 4, the free cross-sectional area of said vessels being larger than that of the pipe conduits before and after the reaction vessel 4. The degradation reaction is terminated by the product being conducted through the inactivation device 18, to which steam is supplied from the pipe 19, and through the pipe conduit 20 serving as inactivation zone. The product thereafter flows through the throttle member 21 and through the expansion chamber 22.

The vessels 6<sup>3</sup>, 6<sup>4</sup> serving for the continuation of the degradation reaction are so designed that their effective volume can be varied. Referring to Figure 3, the vessel 6<sup>3</sup> contains a displacement member 23 which is arranged coaxially thereof and which consists of two sleeves 23<sup>1</sup> and 23<sup>2</sup> closed at one end, the said sleeves engaging telescopically one within the other at their open ends while sealing off their internal space from the free space of the vessel 6<sup>3</sup>. The sleeve 23<sup>1</sup> is secured by means of tubes 24 to the wall of the vessel 6<sup>3</sup>. On the other hand, the sleeve 23<sup>2</sup> is freely movable axially and is guided by a rod 25 extending through the bottom of the vessel 6<sup>3</sup>. It is thus possible for the volume of the displacement member 23 to be varied.

The primarily liquefied starch paste coming from the reaction vessel 4 passes through a pipe 26 into the vessel 6<sup>3</sup> and leaves the latter through a pipe 27. The effective volume of the vessel 6<sup>3</sup> can now be varied by altering the volume of the displacement member 23 for the purpose of adjusting the residence time of the starch paste in the vessel 6<sup>3</sup>. For this purpose, either the rod 25 is moved from outside in its axial direction, or a gaseous or liquid medium is introduced into or released from the interior of

the displacement member 23 through one of the tubes 24. With this method of procedure, it is also possible to use an elastically deformable container as displacement member instead of a displacement member consisting of components displaceable one within the other.

According to Figure 4, the vessel 6<sup>4</sup> is constructed as a tube with a semi-spherical base. The possibility of changing the effective volume of the vessel 6<sup>4</sup> is achieved by a piston-like construction of the upper closure member 28. The piston 28 has a semi-spherical recess on the side facing the liquid for improving the guiding of the liquid. However, it could for example also be constructed as a hollow cone for this purpose. The piston 28 can be displaced axially by a rod 29. The initially liquefied starch paste enters through a pipe 30 opening into the vessel 6<sup>4</sup> at the bottom and leaves the vessel 6<sup>4</sup> through a flexible pipe 31 connected in the middle of the piston 28.

As shown in Figure 2, the installation also comprises various regulating means. One of these regulating means contains a viscometer 32 as measuring element, which can be operated by the rotation principle or which is advantageously formed as a diaphragm traversed at constant velocity by the expanded and deaerated end product. The regulating impulse, which in the second case is the difference of the operative pressures of the said diaphragm, then travels by way of a transmitter 33 either to the rod 25 of the displacement member 23 in the vessel 6<sup>3</sup> or to the rod 29 of the piston 28 in the vessel 6<sup>4</sup>. By suitable adjustment of the effective volumetric capacity of these vessels and thus the reaction period, the progress of the reaction is so controlled by the regulating means 32, 33, 25, 23, or 32, 33, 29, 28 that the viscosity of the end product assumes a prescribed value. The regulating impulse from the viscometer 32 could however also be conveyed by a path not illustrated in the drawing by way of the transmitter 33 and a gas or liquid supply to the tube 24 in the reaction vessel 6<sup>3</sup>. By gas or liquid at suitable pressure being injected into or discharged from the displacement member 23, the effective size thereof and thus the reaction time of the starch produce would be modified.

Alternatively, the regulating impulse of the viscometer 32 can moreover also be conducted through a transmitter 34 to a valve 35 fitted in the steam pipe 5, in order to alter the reaction temperature adjusted by supply of steam in the reaction vessel 4 for the purpose of correcting the viscosity of the end product. Finally, a tank 1<sup>1</sup>, is also provided from which the enzyme solution or suspension necessary for the degradation reaction can be completely admixed, or

a necessary additive can be admixed, by way of a regulating valve 36 or a proportioning pump, with the starch suspension to be treated and in a mixing arrangement 37 of substantially the same type as the reaction vessel 10 in Figure 1. The regulating valve 36 can likewise be adjusted through a transmitter 38 under the influence of the viscometer 32. It is therefore possible in this case for the quantity of the secondary active substance added to be correspondingly adjusted for regulating the properties of the end product.

In addition, a regulating circuit is also provided for adjusting the regulating valve 35 by a thermometer 39 connected after the reaction vessel 4 and measuring the reaction temperature therein through a transmitter 40, and also a regulating circuit for influencing a regulating valve 41 connected into the steam pipe 19 by a thermometer 42 measuring the inactivation temperature through a transmitter 43.

Instead of the viscometer 32, it is also possible to instal in the regulating means a measuring instrument for another physical or even a chemical property of the end product. In particular, a regulating means of the type described can also be used in the production of *d*-glucose, for example in the installation according to Figure 1. The vessel 6<sup>1</sup> or 6<sup>2</sup> serving for the continuation of the degradation reaction would then have to be replaced by a vessel of the type having a variable effective volumetric capacity, for example of the type such as 6<sup>3</sup> or 6<sup>4</sup> in Figure 2. Instead of an instrument measuring the viscosity, it would then be possible to use, as impulse emitter, an instrument for determining the degree of saccharification, for example a colorimeter or a turbidometer. The impulse being sent from the measuring instrument could then be utilised, after suitable amplification, for varying the effective volumetric capacity of the reaction vessels, for varying the enzyme proportioning or for varying the reaction temperature.

The following example is intended to provide further details of the process of this invention as performed using the installation of Figure 2, all percentages are percentages by weight unless otherwise stated.

#### EXAMPLE 3

By mixing 100 kg. of maize starch of normal commercial humidity with 200 litres of tap water, a 30% starch suspension is produced in the tank 1 (Figure 2) 0.2% of an  $\alpha$ -amylase of industrial purity, based on the air-dry starch charge, is added thereto and the pH value is adjusted to a value favourable for the enzymatic conversion. The amylase is stabilised by adding calcium phosphate or phytate. The starch suspension prepared in this way for the conversion is



continuously conveyed through the reaction vessel 4 where it is heated to 90°C. by mixing with steam, and subjected to high imposed stress by impacting and/or shearing forces. The gelatinisation and primary starch liquefaction produces a product with a viscosity of 350 cP. The starch product is conducted through the additional vessel 6<sup>3</sup> or 6<sup>4</sup> having a capacity of about 100 litres and serving for continuing the degradation reaction and remains therein for 5 to 20 minutes, depending on the required degree of degradation of the starch. The counter-pressure built up by the reaction vessel and the necessary bend in the pipe conduit does not exceed 1 atm. gauge in this case. The sugar solution thereafter passes through the device 18, which serves for inactivating the enzyme, and is heated to at least 110°C for feeding in steam at a pressure of to 2 atm. gauge. The sugar solution remains for 30 seconds to 2 minutes in the subsequent pipe section 20 for complete enzyme inactivation. A sugar solution is formed which contains mainly limit dextrans and maltose and which can be processed to starch syrup by decolorisation, elimination of substances causing turbidity and concentration by evaporation.

#### WHAT WE CLAIM IS:—

1. A continuous process for the manufacture of starch decomposition products, particularly *d*-glucose or dextrin, which process comprises the steps of adding a chemically and/or biologically active substance to a starch suspension, passing the mixture continuously through a reaction vessel with temporary acceleration of the flow followed by direct mixing with steam, the added substance becoming active during and directly after the steam passage in a field of action of high imposed stress by impacting and/or shearing forces, and conducting the primarily liquefied starch paste thus obtained through at least one additional reaction vessel for the purpose of continuing the degradation reaction and adjusting required chemical or physical properties, the free cross-sectional area of the additional reaction vessel being larger than that of the pipe conduits before and after the first-mentioned reaction vessel.

2. A process according to claim 1 in which, after flowing through the additional reaction vessel, at least one additional chemically or biologically active substance is fed into the continuously flowing starch product.

3. A process according to claim 1 or claim 2, in which a temperature-sensitive carbohydrase is used as additional active substance.

4. A process according to claim 2 or claim 3 in which the flowing starch pro-

product is cooled before the additional active substance is introduced.

5. A process according to any one of claims 2 to 4, in which the additional active substance is fed in as a solution or suspension in water for the purpose of cooling the flowing starch product.

6. A process according to any one of claims 2 to 5, in which the flowing starch decomposition product, after feeding in the additional active substance, is conveyed for the purpose of intensive mixing of the components through an arrangement in which the components, in the form of a film and with single or multiple deflection and acceleration, are exposed to impacting and/or shearing effects of high frequency.

7. A process according to any one of claims 2 to 6, in which the additional active substance is fed in between two vessels serving for the continuation of the degradation reaction.

8. A process according to claim 1 in which the starch decomposition product, for thermally inactivating the active substances, is mixed under super-atmospheric pressure with steam for the purpose of raising the product to a temperature above 110°C and is relieved of pressure after passing through a vessel for the completion of the inactivation.

9. A process according to claim 1, in which the reaction temperature in at least one of the reaction vessels is adjustable for regulating the properties of the end product.

10. A process according to claim 1, in which the quantity of added active substance is adjustable for regulating the properties of the end product.

11. An installation for effecting the process according to claim 1, comprising a first reaction vessel for the continuous primary liquefaction of starch as a result of the action of a chemically and/or biologically active substance, means for inactivating or destroying this active substance, and a second reaction vessel for continuation of the degradation reaction, said second reaction vessel being connected between said first reaction vessel and said means and having a free cross-sectional area which is larger than the pipe conduits before and after the said first reaction vessel.

12. An installation according to claim 11, in which the said second reaction vessel contains screens or sieves for making the flow uniform and for producing a mixing effect.

13. An installation according to claim 12, in which the free cross-sectional area of the individual apertures of the screen decreases from the inlet side to the outlet side of the vessel.

14. An installation according to claim 11,

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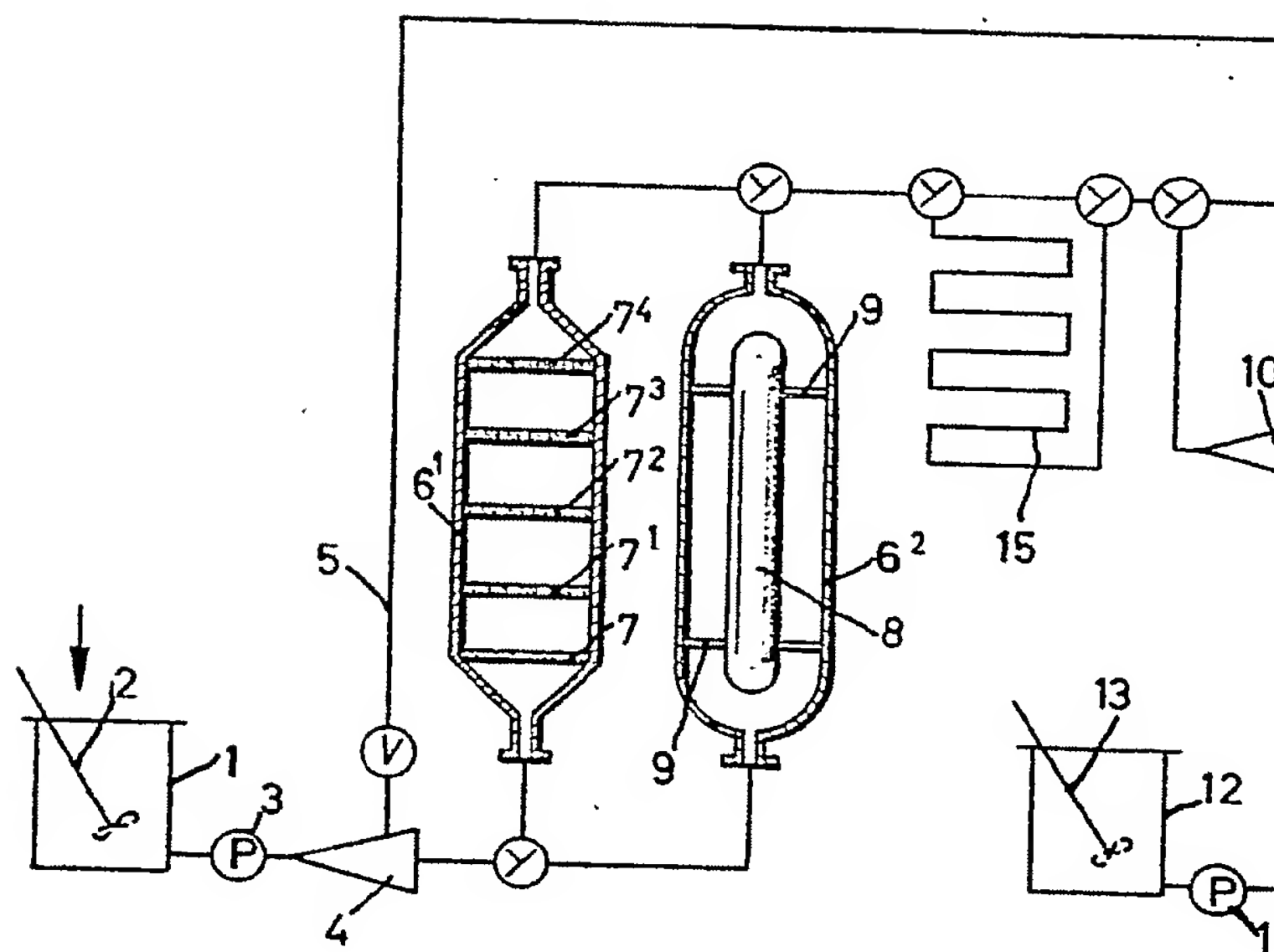
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- in which said second reaction vessel contains a displacement member.
- 5 15. An installation according to claim 11, in which the effective volumetric capacity of the said second reaction vessel is adapted to be altered.
- 10 16. An installation according to claim 14, in which the volume of the displacement member is adapted to be altered.
- 15 17. An installation according to claim 16, in which the displacement member consists of an assembly of two sleeves which are relatively displaceable one within the other and are closed at one end.
- 20 18. An installation according to claim 15, in which said second reaction vessel incorporates a piston-like closure member.
- 25 19. An installation according to claim 15, for the production of dextrin, in which regulating means are provided for adjusting the reaction period during the flow through said second reaction vessel, this regulating means containing a viscometer as a measuring element for the purpose of adjusting the effective volumetric capacity of this reaction vessel.
- 30 20. An installation according to claim 19, in which the viscometer comprises a diaphragm traversed at a constant velocity by the starch decomposition product, and in which the difference in the effective pressure before and after the diaphragm serves for adjusting the effective volumetric capacity of the reaction vessel.
21. An installation according to claim 15 35 for the production of *d*-glucose, in which regulating means are provided for adjusting the reaction period during the flow through said second reaction vessel, these regulating means containing an instrument for determining the degree of saccharification and adjusting the effective volumetric capacity of said reaction vessel.
22. An installation according to claim 21, 40 wherein said instrument is a colorimeter or tubidometer.
23. A continuous process for the manufacture of starch decomposition products substantially as described in any one of the 50 examples herein.
24. An installation for use in the manufacture of starch decomposition products constructed and arranged to operate substantially as described herein with reference 55 to, and as illustrated by, Figure 1 or Figure 2 of the accompanying drawings.
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Fig.1



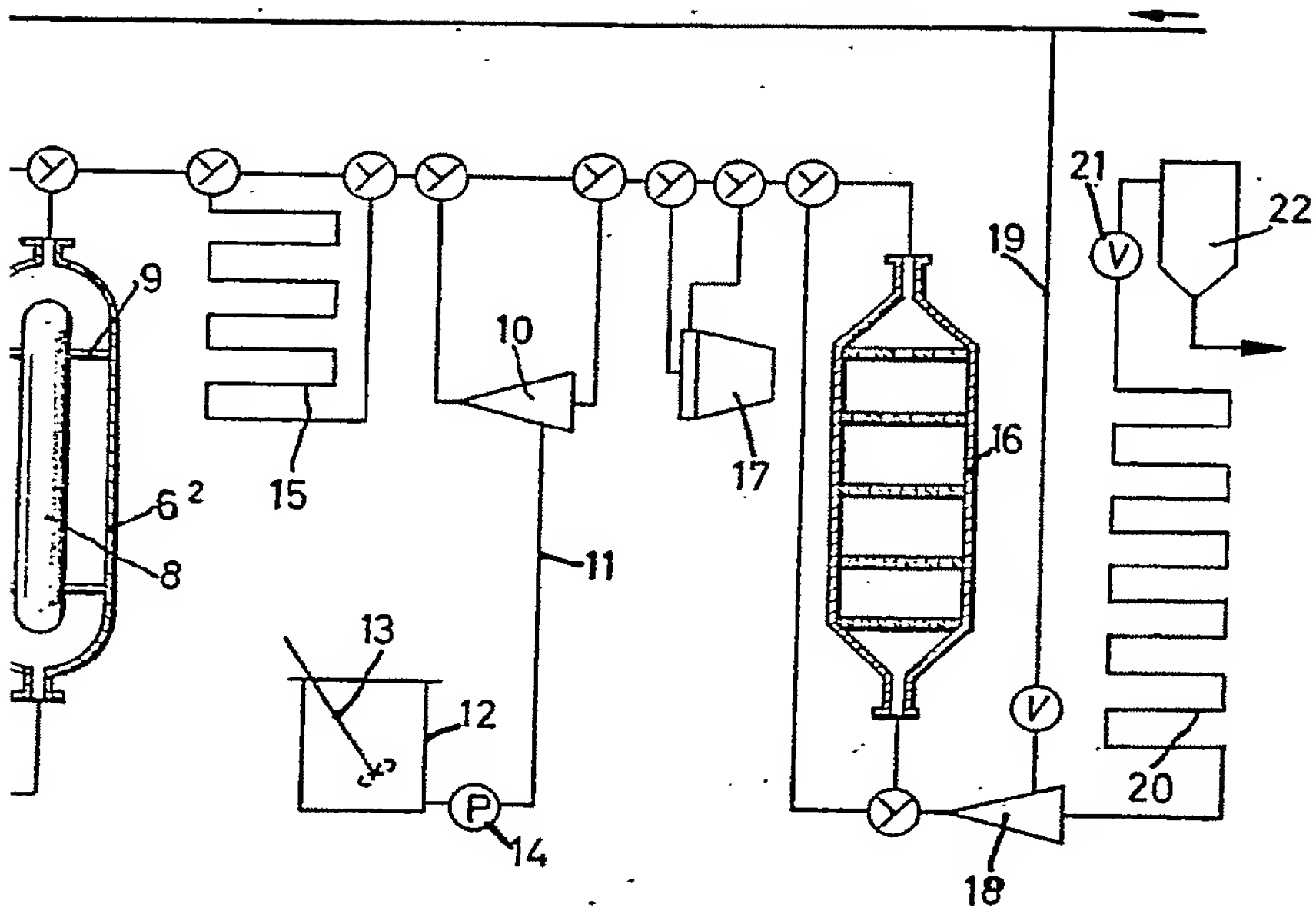




Fig.1

